

# PATENT SPECIFICATION



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## COMPLETE SPECIFICATION.

### Manufacture and Application of Stable Diazo-preparations.

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We, SOCIETY OF CHEMICAL INDUSTRY IN BASEL (also known as Gesellschaft für Chemische Industrie in Basel), of Basle, Switzerland, a body corporate organised according to the laws of Switzerland, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

It is known that diazo-preparations can be made by precipitating a diazo-solution with an aromatic sulphonic acid.

This procedure is not of general application, since many diazo-compounds are not precipitated or only incompletely precipitated by an aromatic sulphonic acid.

This invention achieves in many cases an essential improvement in the separation of the diazo-compound, by conducting the precipitation by means of the aromatic sulphonic acid in presence of a salt of a metal of the second group of the periodic system of the elements, advantageously a salt of magnesium.

The products thus precipitated contain a metal, probably in the form of a double compound, and are characterised by their good solubility, which is remarkable in view of the ease with which they are precipitated. They are very stable and well suited for the production of dyeings by the methods hitherto usual in the so-called ice dyeing or printing.

As to the aromatic sulphonic acids which are available, there may be named the sulphonic acids of the benzene series and of the naphthalene series, such as benzene-sulphonic acids, toluene-sulphonic acids, chlorobenzene-sulphonic acids, chlorotoluene-sulphonic acids, naphthalene-mono and poly-sulphonic acids.

The following Examples illustrate the invention, the parts being by weight:—

#### EXAMPLE 1.

88 parts of 2:5-dichloraniline-hydrochloride are introduced into as little ice as possible (100 parts) and hydrochloric acid, and diazotised by addition of the necessary proportion of nitrite solution. Into the diazo-solution are introduced 80 parts of pulverised magnesium chloride [Price 1/-]

( $MgCl_2 \cdot 6H_2O$ ). After the salt is dissolved the whole is filtered and the clear diazo solution is mixed while stirring with a concentrated solution of 75 parts of sodium 2:7-naphthalenedisulphonate. There is soon formed an intensely bright yellow precipitate. This is filtered, pressed and dried in a vacuum at 30—40° C. When the operation is conducted without the use of magnesium chloride no diazo-compound is precipitated, even by addition of common salt.

The new diazo-compound is relatively freely soluble in water and this solubility can in some cases be increased by addition of acid or an easily soluble neutral or acid salt (compare Specification No. 238,704).

#### EXAMPLE 2.

25 parts of ortho-anisidine are mixed with 48 parts by volume of concentrated hydrochloric acid and the mixture is cooled externally by means of ice, while it is strongly stirred. To the suspension of the hydrochloride thus obtained, with addition of only a little ice or without such addition, there are gradually added 40 parts by volume of a 5N-sodium-nitrite solution. When the diazotisation is complete 21 parts of crystallised magnesium chloride are added and the solution is then filtered. To the clear diazo-solution are added 35 parts of sodium 2:7-naphthalene-disulphonate, either solid or in the form of a concentrated solution. There separates a yellow precipitate, which is filtered and dried in a vacuum at 30—40° C. This product is very stable and very easily soluble in water, from which it may be recrystallised.

#### EXAMPLE 3.

52 parts of ortho-chloraniline are stirred with 100 parts of water and 100 parts by volume of concentrated hydrochloric acid, the suspension is cooled in ice and 80 volumes of 5N-sodium-nitrite solution are gradually dropped in. When diazotisation is complete 82 parts of powdered crystallised magnesium chloride are added, and the solution is then filtered. A boiling hot concentrated solution of 75 parts of sodium 1:5-naphthalene-disulphonate is now added to the clear

diazo-solution. On cooling, there crystallises a nearly white precipitate, which is filtered, freed from liquid by suction or pressure and dried in a vacuum at 30—40° C. The product is very stable and freely soluble in water, from which it can be recrystallised.

In precipitating 2:5-dichloraniline as prescribed in Example 1, calcium chloride may be used instead of magnesium chloride. A precipitate is also obtained if in Example 1 sodium 1:3:6-naphthalenetrisulphonate is substituted for the 2:7-naphthalenedisulphonate.

Similar diazo-preparations may be obtained with other acids and other amines. For example, from magnesium chloride, 2-diazo-4-chloro-1:1'-diphenyl ether and para-toluenesulphonic acid.

**EXAMPLE 4.**

Cotton is padded in known manner with a solution of 12 grams of the  $\alpha$ -naphthylamide of 2:3-oxynaphthoic acid and the goods thus treated are developed with a solution which contains per litre 50 grams of the product obtainable as described in the first paragraph of Example 1, and 1 cc. of acetic acid. There is obtained a yellowish scarlet tint of the known very good properties.

Instead of the  $\alpha$ -naphthylamide, other coupling components may be used, for instance the anilide, the para-chloranilide, the  $\beta$ -naphthylamide of 2:3-oxynaphthoic acid. Also 1-oxynaphthy-

lene-4-phenylketone, an arylide of acetoacetic acid, such as diaceto-acetic-orthotolidide; also instead of the product of Example 1 other preparations obtainable according to this invention may be used.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A manufacture of solid, stable diazo-preparations by precipitating a diazo-compound from a solution thereof by means of an aromatic sulphonic acid in presence of the salt of a metal of the second group of the periodic system of the elements.

2. Diazo-preparations which can be made by the manufacture referred to in claim 1 whenever so made or made by any process which is the obvious chemical equivalent of such manufacture.

3. A process of dyeing wherein the goods to be dyed are treated with a product referred to in claim 2, by the so-called ice dying or printing method.

4. Goods which have been dyed or printed by the process referred to in claim 3.

Dated this 17th day of June, 1930.

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